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(54) Title: PROCESS FOR COATING A PARTICLE WITH A POLYMERIC COATING HAVING UNIQUE DISSOLUTION CHARACTERISTICS

(57) Abstract

A process for coating a solid or composite particle with a protective alkyl cellulose ether polymeric coating which is insoluble in a liquid detergent product but soluble in the wash solution of the liquid detergent product is disclosed. In this process, an alkyl cellulose ether polymer is dispersed in water. The temperature of the water is at least about 60 °C. The alkyl cellulose ether polymer in water dispersion is then cooled to a temperature below at least about 40 °C and a sprayable alkyl cellulose ether polymer in water solution is formed which is then sprayed on the solid or composite particle while the temperature of the sprayable solution is simultaneously maintained below about 40 °C. A coating of the alkyl cellulose ether on the solid or composite particle is formed.

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PROCESS FOR COATING A PARTICLE WITH A POLYMERIC COATING HAVING UNIQUE DISSOLUTION CHARACTERISTICS

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TECHNICAL FIELD

The present invention generally relates to liquid detergent compositions. More particularly, the invention relates to a process for coating particles which are useful in liquid detergent compositions, with a minimum but effective amount of a protective polymeric alkyl cellulose ether coating which remains insoluble in the liquid detergent composition but becomes soluble in the wash solution.

15 <u>BACKGROUND OF THE INVENTION</u>

It has been recognized that consumers of liquid detergent products, such as liquid automatic dishwashing (ADW), liquid hand dishwashing formulas (LDLs), or liquid laundry detergent products prefer that these products having a pleasing color or appearance. Providing a pleasing color to the liquid detergent is one way of improving its appearance. Adding solid particles which are suspended in a liquid detergent composition is another way to improve the aesthetics of the product. Another way is to make the particles colored and match their color to the base color of the liquid detergent composition to create a pleasing combination of colors.

In the past, in an effort to improve the aesthetics of a liquid detergent products, the particles added to these products have been coated with thick waxy or wax-based coatings. This has a major disadvantage of detrimentally causing filming, particularly on glass, stainless steel and plastic surfaces because the water-insoluble waxes melt and are released into the wash solution during the high temperature encountered during an automatic dishwashing process.

It has been desirable to have a process for forming a coating on a particle for a liquid dishwashing product that does not cause a significant amount of unwanted filming on glass, stainless steel and plastic. In view of the foregoing, it has thus been desirable to have a process for coating particles which are useful in liquid detergent compositions for improvement or aesthetics, with a minimum but effective amount of a protective polymeric alkyl cellulose ether coating which remains insoluble in the liquid detergent composition but becomes soluble in the wash solution.

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The inventors of the present invention have discovered a process for coating particles with a minimum but effective amount of a protective polymeric alkyl cellulose ether coating which remains insoluble in the liquid detergent composition but becomes soluble in the wash solution.

The present invention is thus directed to overcome one or more of the problems as set forth before.

SUMMARY OF THE INVENTION

The invention meets the needs above by providing a process for coating a solid or composite particle with a protective alkyl cellulose ether polymeric coating which is insoluble in a liquid detergent product but soluble in the wash solution of the liquid detergent product.

In one aspect of the present invention, the process comprises the following steps. An alkyl cellulose ether polymer is dispersed in water. The temperature of the water is at least about 60 °C. The alkyl cellulose ether polymer and water are present in a weight ratio in a range of from about 1:99 to about 30:70, polymer:water. The alkyl cellulose ether polymer in water dispersion is then cooled to a temperature below at least about 40 °C and a sprayable alkyl cellulose ether polymer in water solution is formed. Then, the sprayable alkyl cellulose ether polymer in water solution is sprayed on the solid or composite particle while the temperature of the sprayable solution is simultaneously maintained below about 40 °C. A coating of the alkyl cellulose ether on the solid or composite particle is formed.

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DETAILED DESCRIPTION OF THE INVENTION

<u>Definitions</u> – By "liquid dishwashing detergent composition", "liquid dishwashing composition" or "liquid dishwashing detergent product" it is meant a detergent composition that is employed in manual (i.e. hand) or automatic dishwashing.

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Without being bound to any particular theory, it has been discovered that when an alkyl cellulose ether polymer is sufficiently hydrated before spraying on the particle or prill, the resultant polymer coating on the particle or prill remains stable, unbroken and undissolved in the liquid dishwashing composition.

The Process

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In the preferred embodiment of the present invention, the hydration is accomplished by the step of dispersing an alkyl cellulose ether polymer in water in a weight ratio in a range of from about 1:99 to about 30:70, polymer:water and forming a sprayable alkyl cellulose ether polymer in water solution. Desirably, the weight ratio of polymer to water is in a range of from about 1:99 to about 20:80, preferably from about 3:97 to about 10:90, more preferably from about

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4:96 to about 6:94, and most preferably about 5:95. The dispersion is accomplished in a contained equipped with a stirring device. The temperature of the water during the stirring is maintained above about 60 °C, desirably, in a range of from about 60 °C to about 90 °C, preferably from about 60 °C to about 80 °C, and most preferably from about 70 °C to about 80 °C.

After dispersing the alkyl cellulose ether in water as described above, the alkyl cellulose ether polymer in water dispersion is cooled to a temperature below at least about 40 °C to assure adequate hydration of the methyl cellulose in the water to form a solution of alkyl cellulose ether in water. In the preferred embodiment, the temperature of sprayable alkyl cellulose ether polymer in water solution is desirably maintained below about 40 °C while spraying this solution on the particle. The temperature of the solution is preferably maintained within a range of from about 30 °C to about 40 °C, more preferably in a range of from about 32 °C to about 38 °C, even more preferably in a range of from about 33 °C to about 37 °C and most preferably at a temperature of about 35 °C.

In the preferred embodiment, the coating is desirably present in an amount in a range of from about 1% to about 25% by weight of the particle, preferably from about 4% to about 10% by weight of the particle, and most preferably, about 5% by weight of the particle. The reduced coating weight levels are desirable so that the least effective coating amount is used in order to accomplish the objective to having a protective coating without unnecessary polymer that might detrimentally contribute to unwanted residue deposition in the washing machine.

In the preferred embodiment, the pH value of the sprayable solution is maintained within a range of from about pH 8.0 to about pH 9.5, and preferably, from about pH 8.5 to about pH 9.0. It is desirable to maintain within the above pH range to allow for full solubilization of the methyl cellulose in water before spraying.

It has been surprisingly found that by using the above process steps, a protective methyl cellulose polymeric coating is formed, which is insoluble in a liquid detergent product but soluble in the wash solution of the liquid detergent product, while at the same time, requiring only about 5% polymer by weight of the particle. This offers an advantage because by using a smaller the quantity of polymer used for coating the prill or particle, there is a reduction in the amount of polymer residue that can potentially re-deposit on the dishware and dishwasher, when the particle dissolves in the wash solution.

Polymeric coating

In the preferred embodiment, the colored polymeric coating is insoluble in a liquid automatic dishwashing detergent composition but soluble in the wash solution. The coating is prepared from alkyl cellulose ethers. Desirably, the alkyl cellulose ethers are methyl cellulose

and hydroxypropyl methyl cellulose (HPMC). Preferably, the coating is prepared from methyl cellulose having a number average molecular weight desirably in a range of from about 5000 to about 100,000, more preferably from about 10,000 to about 20,000, and most preferably, about 14,000. The preferred methyl cellulose is one sold under the trade name Methocel A15LV, and manufactured by Dow Chemicals. Alternatively, the polymeric coating is polyvinyl alcohol (PVA) having a molecular weight, desirably in a range of from about 5000 to about 100,000, and preferably from about 13,000 to about 23,000. The preferred PVA is from about 87% to about 89% hydrolyzed, such as a commercially available product having a trade name Airvol 205.

Accordingly, having thus described the invention in detail, it will be obvious to those skilled in the art that various changes may be made without departing from the scope of the invention and the invention is not to be considered limited to what is described in the specification.

Methods for measuring density are well known to those skilled in the art. Without limiting the scope of the invention, examples of these methods for liquids can be found in Vogels Textbook of Practical Organic Chemistry (Fourth Edition), 1978, published by Longman Inc, New York. A preferred method for measuring the density of a solid helium pycnometry. Helium pycnometry measurement devices are well-known to those skilled in the art and available from the Micrometrics Co.

Examples of typical hand and automatic liquid dishwashing formulas are outlined below. Detailed descriptions of typical hand and automatic liquid dishwashing ingredients can be found in U. S. Pat. No. 6,020,294, issued February 1, 2000, to Getty, et al.; U. S. Pat. No. 5,912,218, issued June 15, 1999, to Chatterjee, et al.; [New LDL3 application will be added]; U.S. Pat. No. 5,415,814 issued 16 May 1995, to Ofosu-Asante et al. All of these references are hereby incorporated by reference.

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	EXAMPLE A				
Ingredient (weight % active)	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>	<u>E</u>
Sodium Tripolyphosphate	17.5	16.0		8.0	16.0
Potassium Tripolyphosphate			16.0	8.0	16.0
Sodium Silicate	6.0	1.0			
Potassium hydroxide	3.6	5.6	5.6	1.0	1.0
Sodium hydroxide	2.0	2.0	2.0		
Polyacrylate polymer	1.0				
Nonionic surfactant		0.5	0.8	0.7	1.5

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	Sodium hypochlorite	1.15				
	Polyacrylate polymer thickener	1.5	1.5	1.5	1.5	1.0
	Solid Prill	0.5	0.5	0.5	0.5	0.7
	Protease enzyme					0.01
5	Water and minors	Bal.	Bal.	Bal.	Bal.	Bal.
	TOTAL	100.0	100.0	100.0	100.0	100.0

EXAMPLE B

Ingredient	<u>F</u>	<u>G</u>	<u>H</u>
Ammonium C_{12-13} alkyl ethoxy(1)	28.0	27.0	26.0
sulfate			
Coconut amine oxide	4.5	6.0	2.5
Alcohol Ethoxylate C ₈ E ₁₁	5.0		5.0
Ammonium xylene sulfonate		4.0	4.0
Ethanol	4.0	4.0	4.0
Magnesium chloride	3.3	3.3	3.3
Amylase enzyme			0.001
Prills	1.0	0.5	1.0
Thixcin	0.5	0.1	
Water and minors	B	alance	

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WHAT IS CLAIMED IS:

1. A process for coating a solid or composite particle with a protective alkyl cellulose ether polymeric coating which is insoluble in a liquid detergent product but soluble in the wash solution of the liquid detergent product, characterized by the steps of:

dispersing alkyl cellulose ether polymer in water wherein the temperature of said water is at least 60 °C, and wherein said alkyl cellulose ether polymer and said water are present in a weight ratio in a range of from 1:99 to 30:70, polymer:water;

cooling said alkyl cellulose ether polymer in water dispersion to a temperature below at least 40 °C and forming a sprayable alkyl cellulose ether polymer in water solution;

spraying said sprayable alkyl cellulose ether polymer in water solution on said solid or composite particle while simultaneously maintaining the temperature of said sprayable solution below 40 °C; and forming a coating of said alkyl cellulose ether on said solid or composite particle.

- 2. The process according to claim 1, including dispersing said alkyl cellulose ether polymer in said water in a weight ratio in a range of from 3:97 to 10:90, polymer:water.
- 3. The process according to any of claims 1-2, including dispersing said alkyl cellulose ether polymer in said water in a weight ratio in a range of from 4:96 to 6:94, polymer:water.
- 4. The process according to any of claims 1-3, wherein the temperature of the water during said dispersion step is maintained in a range of from 60 °C to 90 °C.
- 5. The process according to any of claims 1-4, wherein the temperature of the water during said dispersion step is maintained in a range of from 60 °C to 80 °C.
- 6. The process according to any of claims 1-5, including maintaining the temperature of said sprayable solution within a range of from 33 °C to 37 °C.
- 7. The process according to any of claims 1-6, wherein said alkyl cellulose ether is methyl cellulose.

- 8. The process according to any of claims 1-7, wherein said coating is present in an amount in a range of from 1% to 25% by weight of said particle.
- 9. The process according to any of claims 1-8 including the step of maintaining the pH value of said sprayable solution in a range of from pH 8.0 to pH 9.5.
- 10. A liquid dishwashing detergent composition characterized by less than 50% free water and from 0.02% to 2.5% of the solid or composite particles prepared according to the process of claim 1.